



UNIVERSITI PUTRA MALAYSIA

**CARBOXYMETHYLATION OF SAGO STARCH AND SAGO WASTE
AND THE FORMATION OF CARBOXYMETHYL STARCH-
HYDROGEL VIA IRRADIATION TECHNIQUE**

NORHAZLIN BINTI ZAINUDDIN

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By

NORHAZLIN BINTI ZAINUDDIN

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
in Fulfilment of the Requirement for the Degree of Master of Science**

January 2003



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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January 2003

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Faculty : Science and Environmental Studies

Carboxymethyl starch (CMS) and carboxymethyl sago waste (CMSW) were synthesized in completely heterogeneous conditions as a product of the reaction of sago starch or sago waste and sodium monochloroacetate ($\text{ClCH}_2\text{COONa}$) in isopropanol and sodium hydroxide (NaOH). The influence of various parameters on the degree of substitution (DS) was determined. The optimum conditions obtained for CMS were as follows: NaOH concentration, 20 % w/v; reaction time, 1 h; reaction temperature, 55°C ; and amount of $\text{ClCH}_2\text{COONa}$, 1.5 mole. Meanwhile the optimum conditions for carboxymethylation of sago waste were as follows: NaOH concentration, 25 % w/v; reaction time, 2 h; reaction temperature, 55°C ; and amount of $\text{ClCH}_2\text{COONa}$, 2.0 mole. The DS and reaction efficiency (RE) under optimum conditions for CMS were 1.05 and 85.9 %, respectively, whereas for CMSW were 1.04 and 79.5 %, respectively.

Solubility test, and analyses such as Fourier transform infrared (FTIR) spectroscopy, differential scanning calorimetry (DSC), X-Ray Diffraction (XRD) analysis, thermogravimetric analysis (TGA) and scanning electron microscopy (SEM) were used to characterize the CMS and CMSW. All CMS samples except CMS10a, dissolved in water, whereas for CMSW samples dissolved partially in water. The infrared spectra of CMS and CMSW showed additional peaks at 1590 cm^{-1} and 1412 cm^{-1} , indicating the presence of $-\text{COO}^-\text{Na}^+$ group. DSC of CMS showed an absence of endothermic peak of native sago starch. XRD studies indicated that the degree of crystallinity of CMS and CMSW were much lower than that in native sago starch and sago waste. TGA studies revealed that the decomposition temperatures of native sago starch and sago waste were different from CMS and CMSW. SEM studies indicated that the surface of CMS and CMSW were rough and grooved compared to smooth surface of sago starch. The CMS-hydrogel was obtained by using irradiation technique and the influence of various parameters on the cross-linking process was determined. The highest value of gel content was obtained at 80 % CMS concentration; irradiation dose, 40 kGy; and DS, 1.05. The swelling of CMS-hydrogel in water and various types of medium decreased as the dose and CMS concentration as well as the DS of CMS increased. The swelling at equilibrium decreased in the order; $\text{H}_2\text{O} > \text{NaCl} \approx \text{NaOH} > \text{HCl}$.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

**PENGKARBOKSILAN KANJI SAGU DAN HAMPAS SAGU DAN
PEMBENTUKAN KARBOKSIMETIL KANJI-HIDROGEL MELALUI TEKNIK
PENYINARAN**

Oleh

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Januari 2003

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Karboksimetil kanji (KMK) dan karboksimetil hampas sagu (KMHS), disintesis dalam keadaan heterogen sebagai hasil daripada tindak balas kanji sagu atau hampas sagu dan natrium monokloroasetat ($\text{ClCH}_2\text{COONa}$) dalam isopropanol dan natrium hidroksida (NaOH). Parameter yang mempengaruhi darjah penukargantian, DP, telah dikenalpasti. Keadaan optimum yang diperolehi bagi KMK adalah seperti berikut: kepekatan NaOH , 20 % berat/isipadu; masa tindak balas, 1 jam; suhu tindak balas, 55°C , amaun $\text{ClCH}_2\text{COONa}$, 1.5 mol. Manakala keadaan optimum untuk pengkarboksimetilan bagi hampas sagu adalah seperti berikut: kepekatan NaOH , 25 % berat/isipadu; masa tindak balas, 2 jam; suhu tindak balas, 55°C , amaun $\text{ClCH}_2\text{COONa}$, 2.0 mol. DP dan peratus kecekapan tindak balas pada keadaan optimum bagi KMK masing-masing adalah 1.05 dan 85.9 %, manakala untuk KMHS masing-masing adalah 1.04 dan 79.5 %.

Ujian kelarutan, analisis spektroskopi inframerah transformasi Fourier, kalorimetri pengimbasan pembezaan, pembelauan sinar-X, termogravimetri, dan mikroskop pengimbasan elektron telah digunakan untuk mencirikan KMK dan KMHS. Kesemua sampel KMK, kecuali CMS10a, larut dalam air, manakala kesemua sampel KMHS separa larut di dalam air. Spektra inframerah bagi KMK dan KMHS menunjukkan terdapat puncak tambahan pada 1590 cm^{-1} dan 1412 cm^{-1} mengesahkan kehadiran kumpulan $-\text{COO}^-\text{Na}^+$. Kalorimetri pengimbasan pembezaan bagi KMK menunjukkan tiada puncak endotermik seperti yang terdapat pada kanji sagu. Kajian pembelauan sinar-X pula menunjukkan darjah penghabluran bagi KMK dan KMHS jauh lebih rendah berbanding kanji sagu dan hampas sagu. Kajian termogravimetri membuktikan bahawa suhu penguraian kanji sagu dan hampas sagu adalah berbeza daripada KMK dan KMHS. Kajian mikroskop pengimbasan elektron pula menunjukkan bahawa permukaan KMK dan KMHS kasar dan bergerutu berbanding dengan permukaan kanji sagu yang licin. Hidrogel-KMK diperolehi hasil daripada teknik penyinaran dan parameter-parameter yang mempengaruhi proses tautsilang dikenalpasti. Nilai tertinggi bagi kandungan gel diperolehi daripada KMK berkepekatan 80 % berat/isipadu, dos penyinaran , 40 kGy, dan DP 1.05. Pengembangan bagi hidrogel-KMK dalam air dan berbagai jenis media menunjukkan penurunan apabila dos penyinaran, kepekatan KMK dan juga DP bagi KMK meningkat. Pengembangan pada keseimbangan menurun dalam urutan; $\text{H}_2\text{O} > \text{NaCl} \approx \text{NaOH} > \text{HCl}$.

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I certify that an Examination Committee met on 31st January 2003 to conduct the final examination of Norhazlin binti Zainuddin on her Master of Science thesis entitled "Carboxymethylation of Sago Starch and Sago Waste and the Formation of Carboxymethyl Starch-Hydrogel via Irradiation Technique" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:


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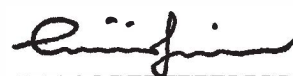
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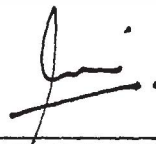


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DECLARATION

I hereby declare that the thesis is based on my original work except for the quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.



NORHAZLIN BINTI ZAINUDDIN

Date: 7 Dec 2003

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LIST OF ABBREVIATIONS

AGU	Anhydroglucose unit
CMC	Carboxymethyl cellulose
CMS	Carboxymethyl starch
CMSW	Carboxymethyl sago waste
DS	Degree of Substitution
$\text{ClCH}_2\text{COONa}$	Sodium Monochloroacetate

CHAPTER I

INTRODUCTION

Polysaccharides such as starch and cellulose play important role in the polymer research because they are from natural sources and biodegradable. Both of these natural products deserve a special position among the industrially used raw materials. Polysaccharides are used in the food industry because they are widely available, usually at low cost and non-toxic. Their use in non-food applications depends on unique special properties they provide, often at costs below those of synthetic polymers.

Modification and derivatization are two techniques applied on cellulose and starch, which are commonly used to widen the applications of these materials in industrial fields. Derivatization such as etherification and esterification as well as modification such as crosslinking and grafting may produce distinct changes in starch and cellulose properties and generally produce polymers with useful properties. Carboxymethyl starch (CMS) and carboxymethyl cellulose (CMC) are among important derivatives, which are readily soluble in water. They are used in paper, textile processing detergents protective coating as well as in the food, pharmaceutical, and cosmetic industries (Brandt, 1986).

Another important modification technique for starch and cellulose is cross-linking. When these materials are reacted with derivatizing agents containing multiple reactive sites, two starches or cellulose polymer chains can share the same substituent and cross-linking occur. Cross-linking can also occur by irradiation technique, which has several advantages such as no catalyst or additives are required and solves the problem of sterilization of the products. Cross-linked starch or cellulose is used when a stable, high viscosity starch or cellulose paste is needed and particularly when the dispersion is to be subjected to high temperature, shear or low pH (Rutenberg and Solarek, 1984).

Nowadays, the potential combination of crosslinkages, depolymerizations and derivatizations of starch and cellulose increase the utilization of these materials. For example, cross-linking the carboxymethyl cellulose by adding small amounts of a second bifunctional reagent during etherification produces highly water absorbing carboxymethyl cellulose. This makes them useful for application in hygienic materials such as diapers or tampons (Brandt, 1986)

In Malaysia, the sago palm has the greatest potential to be a producer of starch. This palm can grow well in peat swamps unlike other crops. It is also the only crop that is capable of giving sustainable economic return on peat swamps in Sarawak. The palm is immune to floods, drought, fire and strong winds. Sago palms continually produce suckers, which in turn grow into adult palms. There is no necessity for replanting and eliminates the need for recurring expensive establishment costs, after every harvest of the adult palms. The extraction process of starch from sago only involves the use of water. The waste products left behind

are the sago bark, the pith and wastewater. The wastewater is discharged back to the rivers, whereas the sago pith can be mixed with supplementary food materials and used as animal feed. Some researchers used the sago bark in the making of particle board (Chew et al., 1999).

In this study, the carboxymethylation of starch and waste product from sago palm was carried out. The modification of these materials gives useful properties and information that can meet the industrial demand.

Significance of the Study

Recently, the increasing demands of plastic materials in industries become a major concern to the environment. Most of plastic materials are non-biodegradable, petrochemical based and do not decompose. The major problems of these materials are not only because of the uncertainties in availability of sufficient petroleum feed stocks, but also the large amount of waste and by product, which is potentially polluting the environment.

In recognition of these problems, interest has increased in the use of natural polymers as extenders for petrochemical plastics. These natural polymers can be found at low cost, and has been known to have the potential to biodegrade. The environmental problem caused by petrochemical plastics and the problems of limited and uncertain supply of petroleum can be solved by alternatively using natural polymers such as starch and cellulose (Otey and Doane, 1984).

In Malaysia, one of potential resources of natural polymers is sago palm. It consists a large amount of starch in its trunk. Sago starch has several advantages such as cheaper price compared to other types of starch, ease to gelatinize and molded (Ahmad et al., 1999). The waste product from the extraction of starch also consists a large amount of cellulose. These two materials, starch and cellulose from sago palm can be use widely as substitute for synthetic polymers. The glycosidic linkages bonding polysaccharide ring are susceptible to biodegradation by microorganisms and hydrolytic enzymes (Wach et al., 2000).

Derivatization and modification of starch and waste from sago promise broad prospects in the industries due to their useful properties. Hence, their environmental friendly nature will solve the major problem of environmental pollution. It will also enhance the growth of sago planting in swamp environment, which is an area that has not been fully utilized.

Objectives of the Study

The objectives of this study are as follows:

1. To prepare carboxymethyl sago starch (CMS) and carboxymethyl sago waste (CMSW) and optimize the reaction conditions, which yields to maximum degree of substitution.
2. To characterize the CMS and CMSW by Fourier Transform Infrared Spectroscopy (FTIR), Differential Scanning Calorimetry (DSC), Thermogravimetry Analysis (TGA), X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM).
3. To prepare hydrogel from CMS by using electron beam irradiation and to optimize the conditions of cross-linking which leads to maximum gel content.
4. To study the swelling properties of the CMS hydrogel in various solvents.

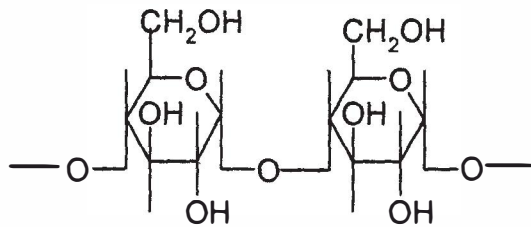
CHAPTER II

LITERATURE REVIEW

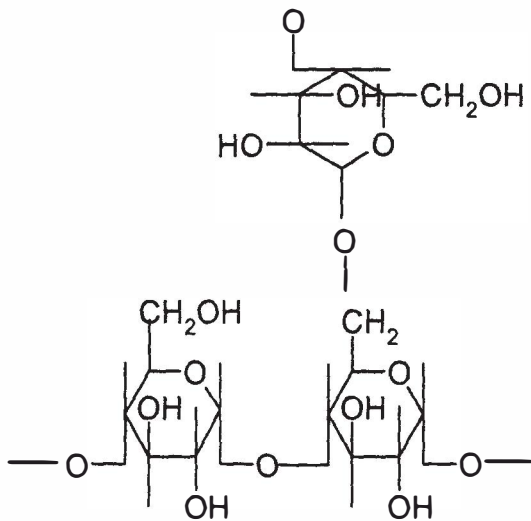
Starch

Starch, $(C_6H_{10}O_5)_n$, the principal reserve polysaccharide in plants, is found widely distributed in nature. It is a natural, biodegradable polymer and the most abundant worldwide commodity. Starch can be found in all organs of higher plants such as seeds, tubes and roots. Most starch is produced from corn, wheat, tapioca, potato, sago and others sources. Starch from each plant variety and species is slightly different in physical and chemical properties, and is synthesized in the form of tiny water-insoluble granules (McCready, 1978).

Most of starches are mixtures of two polymers, amylose and amylopectin as shown in 1 and 2. Amylose is a linear polymer of $(1 \rightarrow 4)$ – linked - α - D- glucopyranosyl unit, whereas amylopectin is a highly branched molecule, consisting of short chains of $(1 \rightarrow 4)$ – linked - α - D- glucopyranosyl unit with $(1 \rightarrow 6)$ – linked branches. In common starch, the amylose constitutes about 20-30% of the starch content, depending on the starch type. Starch occurring in nature is highly hydroxylated, with three hydroxyl groups per anhydroglucose repeating unit at C-2, C-3, and C-6 positions. The reactive hydroxyl groups can be oxidized, derivatized and cross-linked (McCready, 1978).



1. Amylose



2. Amylopectin

Starch occurs in plants in the form of white, sand like granules which may vary in diameter from 2-150 μm . Scanning electron microscopy shows that starches vary in shape from highly angular in rice and corn, to round and oval in wheat and tapioca, or egg-shaped in sago and potato. Microscopic examination with polarized light reveals a birefringence, which along with X-ray diffraction, is an evidence of

semicrystallinity of the granule. Cereal starches give an A-type X-ray pattern, tuber starches a B-type pattern, and a few starches give an intermediate diffraction pattern, the C-type (Jarowenko, 1970).

Although starch granules are insoluble in water at ambient temperature, they imbibe water reversibly and swell slightly. In hot water, a larger irreversible swelling occurs producing gelatinisation. Gelatinisation takes place over a discrete range of temperatures that depends on starch type (Whistler and Daniel, 1983). During heating process, starch granules undergo irreversible changes such as crystallite melting, loss of birefringence, irreversible swelling, and leaching of amylose produced by collapse of molecular orders (BeMiller, 1993).

Cellulose

Cellulose is one of the important and most abundant organic raw materials beside starch. Green plants, both marine and terrestrial, and also a small fraction by few bacteria, animals and fungi produce cellulose. Most cellulose are produced from cotton, jute, ramie, and kenaf. Cellulose is biologically degradable, thus, ecologically beneficial. Cellulose products such as paper or cellulosic textiles will decompose and eventually form valuable humus (Krassig et al., 1985).

Cellulose has the same empirical formula as starch, $(C_6H_{10}O_5)_n$, but the D-glucopyranosyl units are linked in the β - D- (1 \rightarrow 4) configuration. This configuration

permits an extended chain enabling molecular alignment in crystallite and provides reduced solubility (Young, 1984). X – ray diffraction and other evidence indicate that cellulose is partly crystalline and partly disordered. Within the elementary fibrils there is presumably crystalline regularity in the molecular arrangement in some regions and disorder in others. In the crystalline regions the cellulose molecules are arranged in ordered lattices, in which the hydroxyl groups are bonded by strong secondary forces (Ekman et al., 1992). The relative amounts of disordered and crystalline cellulose have a marked effect on such fiber properties as equilibrium moisture content, strength, flexibility, and reactivity (Turbak et al., 1978).

Cellulose is relatively hygroscopic. It swells but insoluble in water or dilute acids. Caustic solutions cause extensive swelling and dissolution of low molecular mass portion. Cellulose is non-melting, and the thermal decomposition starts at 180°C, and the ignition point is more than 290°C. The chemical character of cellulose is determined by the sensitivity of the β -glucosidic linkages between the glucose-repeating units to hydrolytic attack and the presence of three reactive hydroxyl groups in each of the glucopyranose units. These reactive hydroxyls like in the starch molecules are able to undergo exchange, oxidation, and substitution reactions, such as esterification and etherification (Krassig et al., 1985).

Besides being able to undergo chemical reaction, these three hydroxyls are also able to interact with one another forming valence bonds. There are two possibilities for the hydroxyl groups in the cellulose molecule to form hydrogen bonds; i) by

interaction between among suitably positioned hydroxyls in the same molecule, which is called intramolecular hydrogen bonding, and ii) by interaction between hydroxyl groups in neighbouring cellulose molecules known as intermolecular hydrogen bonding. The intramolecular hydrogen bonding is the main cause of the relative stiffness and rigidity of the cellulose molecule, therefore gives high viscosity in solution, high tendency to crystallize, and able to form fibrillar strands (Krassag, 1992).

Sago

Sago palm (*Metroxylon* spp) is a palm species and belongs to the order Arecales, family Palmae, subfamily Calamoidae, tribe Calamae, sub tribe Metroxylinae and genus *Metroxylon*. The origin of sago palm is believed to be the area extending from Moluccas of Indonesia to New Guinea. At present, sago palm grows widely in Southeast Asia and Oceania. The palm occurs between 10° S and 10° N latitudes, and up to an elevation of 700m or 1,000m. This palm is one of the dominant species occurring in tropical peat swamp rain forest and can also grow in dry land (Hisajima, 1995).

The biggest sago areas in Malaysia are to be found in the state of Sarawak. Sarawak is now the world's biggest exporter of sago, exporting annually about 25,000 to 40,000 tons of sago products to Peninsular Malaysia, Japan, Taiwan, Singapore, and other countries (Chew et al., 1999). Sago is a very high yield starch crop that does